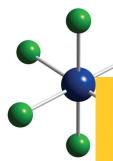
Los Alamos National Laboratory ACTINIDE RESEARCH QUARTERLY

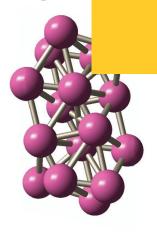


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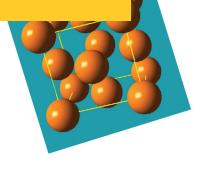


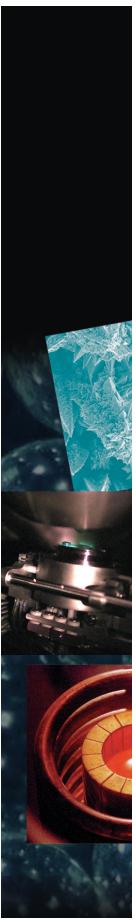
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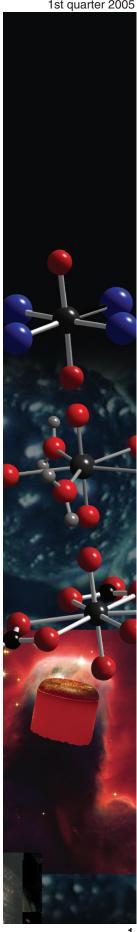
ARQ: 10 years and counting

his issue of *Actinide Research Quarterly* marks the beginning of the publication's second decade. Throughout the past ten years, ARQ has showcased progress in actinide science at Los Alamos, and especially in the Nuclear Materials Technology (NMT) Division, in such areas as process chemistry, metallurgy, surface and separation sciences, atomic and molecular sciences, actinide ceramics and nuclear fuels, characterization, spectroscopy, analysis, and manufacturing technologies. ARQ is instrumental in educating our colleagues—whether internal, national, or international—on the vital research being done at Los Alamos to support the nation's needs in national security, nonproliferation and energy security, and environmental stewardship.

ARQ was launched in December 1994 under the guidance of NMT Division's chief scientist, K.C. Kim, who saw a need in the division for a scientific periodical to keep staff apprised of new scientific and technical developments relevant to NMT's mission. Over the years ARQ has grown in size, distribution, and scope. The current distribution of almost 3,500 goes not only to NMT Division employees and selected colleagues at the Lab but also to funding agencies in Washington, D.C.; U.S. House and Senate special committees; college and university nuclear engineering, chemistry, and physics departments; and national and international peers. The publication can also be read on the World Wide Web at http://www.lanl.gov/arq/.

When Kim retired several years ago, David L. Clark and Gordon Jarvinen of the Seaborg Institute took over as the magazine's scientific advisors. Ann Mauzy and Meredith Coonley of Communication Arts and Services (IM-1) have served as editors. Mauzy joined the magazine's staff with the second issue and continued for almost six years. Coonley took over with the first issue of 2001. One member of the staff has remained constant since ARQ's first eightpage issue: illustrator and designer Susan Carlson, also of IM-1. Carlson is primarily responsible for the evolution of the publication's design from a simple black-and-white layout to the ARQ's current four-color design.

The next decade of science is bound to be an exciting one as the nation's nuclear strategy evolves. A short list of new research endeavors includes the Advanced Fuel Cycle Initiative; the 2010 Initiative to build the first new reactor in the United States in 30 years; the Gen IV program to design the next generation of reactors; and the Nuclear Hydrogen





Initiative, which raises the possibility of using nuclear power to make hydrogen fuel.

NMT Division and the Lab will also continue to make significant contributions to stockpile stewardship and weapons engineering and manufacturing in support of our national security mission. In lieu of underground testing, the small-scale experimentation and integrated tests that NMT Division conducts in collaboration with other divisions will continue to play an important role in certifying newly manufactured weapons. Key to understanding and predicting stockpile aging are our efforts in the Pit Surveillance and Enhanced Surveillance Programs, and work is beginning in actinide surface science, metallurgy, and physics that promises to provide new understanding to poorly understood phenomena.

I want to commend the staff of *ARQ* for producing a quality publication that I consider to be the flagship of NMT Division's communications efforts. I encourage readers to continue to support the magazine by suggesting topics or contributing articles. In the coming decade *Actinide Research Quarterly* will continue to play an important role in educating about the useful role actinides play in our world and in attracting a new generation of actinide scientists to Los Alamos.

-Steve Yarbro, NMT Division Director



Alpha-plutonium's elastic properties:

Windows into new fundamental understanding

ur accurate measurements of alpha (α)-plutonium's low-temperature elastic constants yield insights into α -plutonium's unusual physical properties, into delta (δ)-plutonium's properties, and into actinide properties in general. Delta-plutonium possesses a four-atom face-centered cubic unit cell (see the article on page 9). Alpha-plutonium possesses a sixteen-atom monoclinic unit cell. (Roughly, we can describe monoclinic as a brick-shaped object sheared along one brick surface.) We focus our discussion on three elastic constants: the bulk modulus B (which reflects resistance

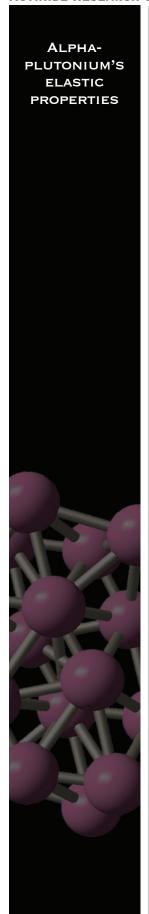
to volume change caused by pressure), the shear modulus G (which reflects resistance to shape change caused by a shearing force), and Poisson ratio (a simple function of the G/B ratio), which, better than any elastic constant, reflects interatomic-bonding type. The bulk modulus joins the binding energy and volume to form the triumvirate of cohesion properties of a crystalline solid.

Although at least eleven measurements were reported of α -plutonium's ambient-temperature bulk modulus, only one very-low-temperature report exists. That study reported not the bulk modulus, but the Young and shear moduli, from which the bulk modulus was computed. The eleven previous measurements, excluding outliers, averaged 50 ± 5 gigapascals (GPa). We found 54.6 ± 0.2 .

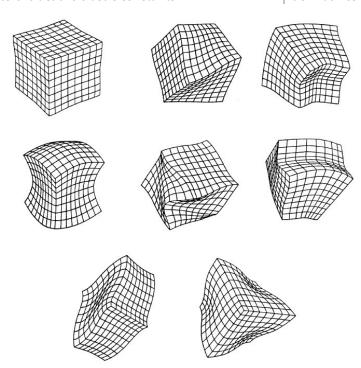
To determine these three fundamental elastic properties, we used resonant-ultrasound spectroscopy (RUS) to measure the normal vibrational modes of a polycrystalline (quasiisotropic) parallelepiped-shape specimen. Crudely speaking, we look for the natural macroscopic vibration frequencies of a simple-shape object, analogous to ringing a bell.

Alpha (α)-plutonium's sixteen-atom monoclinic unit cell consists of two eight-atom layers, both highly distorted hexagonal networks. With only four symmetry elements, this may be the least-symmetrical unit cell among the elements. Delta (δ)-plutonium's face-centered cubic unit cell possesses forty-eight symmetry elements, the highest-possible symmetry. The crystallographic mechanism for the δ - α transformation remains disputed.





The figure below shows an example of each type of mode, symmetric or antisymmetric, across the three spatial directions. The RUS method is extremely accurate, convenient, and it provides all the elastic moduli in one measurement, but requires complex computations to extract the elastic constants.



These eight figures are computer-generated examples of vibration normal-modes of a rectangular parallelepiped. Mode shapes depend on specimen shape, mass density, and anisotropic (fourth-order-tensor) elastic coefficients, as well as on the relationship between the crystal axes and the macroscopic specimen axes. In principle, we can use any regular shape: parallelepiped, cylinder, sphere, or ellipsoid. We require sophisticated algorithms to extract the elastic-stiffness coefficients from the normal-mode frequencies.

Some principal results of our measurements appear in the two diagrams on the next page. The curves represent a function derived from the energy of an ensemble of thermally populated quantized vibrations (the phonon frequencies). Because elastic moduli are second derivatives of the energy with respect to

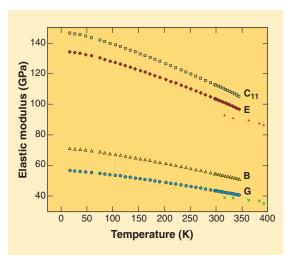
deformations, an elastic modulus C(T) changes with temperature T according to C/T = C(0)[1 - K < E>], where <E> denotes the average oscillator energy and K a constant that depends on crystal structure. Instead of using the exact phonon frequencies, a successful approximation is to choose a single average oscillator energy (the Einstein-oscillator approximation) so that

$$C(T) = C(0) - s / [\exp(t/T) - 1].$$
 (1)

This function and approximation are derived from fundamental physics of elasticity and apply to a wide material variety, ranging, for example, from diamond to the negative-thermal-expansion insulator zirconium tungstate to the metal copper. For the three parameters needed to

fit this function to our measured bulk modulus, we obtained C(0) = 70.9 GPa, s = 11.3 GPa, and t = 158.8 kelvins (K). For this model, $t \approx \Theta_E = (3 / 4)\Theta_D$; Θ_E and Θ_D being the Einstein and Debye characteristic temperatures.

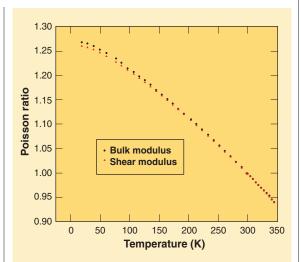
Our measurements reveal that α -plutonium, the stiffest allotrope, shows larger elastic-stiffness changes with temperature than the much softer allotrope, δ -plutonium, which is an unexpected result. Stiffer materials usually show the weakest softening upon warming. Our measurements also reveal that a familiar, but usually ignored, elastic parameter—the



The variation of several elastic constants with temperature is shown for alpha (α) -plutonium. The figure includes four elastic constants: longitudinal modulus C_{11} , Young (extension) modulus E, bulk modulus B, and shear modulus G. All show smooth behavior consistent with an Einstein-oscillator model. The + and \times symbols denote values from older measurements by others.

dimensionless Poisson ratio (v)—shows temperature independence: an exceptional event.

A simple electron-gas viewpoint accounts for the unusual softening of the bulk modulus and shear modulus and the invariance of the Poisson ratio v. For this model, elastic stiffnesses decrease as electrons change from itinerant to localized, from the free-electron gas to electrons fixed at ion positions, as we describe below. Applying this viewpoint predicts approximate divalency for plutonium, implying a 5f 6 localized-electron configuration. (The most advanced theories suggest either 5f 4 or 5f 5 .)



The relative bulk and shear moduli, B and G, versus temperature are shown for alpha (α)-plutonium. Unusually large (30 percent) changes occur, which we attribute to itinerant-to-localized electron transitions during warming. The unusual same percentage changes mean a constant B/G ratio and therefore a constant Poisson ratio ν ; again, results expected for itinerant-to-localized electron transitions. The slight additional softening above about 325 kelvin (K) may represent premonitory behavior for the α - β transition near 400 K.

For the bulk modulus at zero temperature, we obtained 70.9 ± 0.05 GPa, much higher than all previous measurements except one. About a dozen theoretical estimates give an average value of 93 ± 48 GPa, numbers much too high.

From the high-temperature slope dB/dT, we can estimate a Gruneisen parameter γ using the following relationship:

$$dB/dT = -3k\gamma (\gamma + 1) / V_a. \tag{2}$$



ALPHA-PLUTONIUM'S **ELASTIC PROPERTIES**

Here, k denotes the Boltzmann constant and V_a atomic volume. Substitution gives $\gamma = 5.1$. The Gruneisen γ comes from an important approximation in condensed-matter physics and can be thought of as the volume dependence of the elastic stiffnesses. In diamond, there is little pressure dependence of the stiffnesses; so γ is small; in lead, a "soft" material, γ is large. There exists an alternative approach to estimating the Gruneisen parameter. We use the following relationship:

$$\widetilde{B} - B_0 = 3k\Theta_E \gamma(\gamma + 1)/2V_a. \tag{3}$$

Here \widetilde{B} denotes the harmonic bulk modulus obtained by extrapolating the B(T) curve linearly to zero temperature. Substitution gives $\gamma = 5.1$. The handbook value from lattice-specific heat is 6.8. However, as we describe later, we believe the best estimate of Gruneisen's parameter equals ~3.5. The higher apparent values from our measurements arise from electron localization-delocalization during temperature change.

Zero-temperature elastic constants provide the best estimate of the Debye characteristic temperature Θ_D . Since Einstein's seminal lattice-vibration studies, many authors calculated the Debye temperature from the bulk modulus. However, Θ_D depends much more on the shear modulus. We use a relationship given by Kim and Ledbetter:

$$\Theta_D = 2933.22 v_m / V_a^{1/3}. \tag{4}$$

Here, v_m denotes mean sound velocity obtained from the average $\langle 1 / v_i^3 \rangle$. This approach gives $\Theta_D = 205$ K. In Equation (4), units on v_m are cm/ μ s and on V_a , \dot{A}^3 . Although Θ_D (elastic) should equal Θ_D (specific-heat) at zero temperature, our value exceeds considerably most specific-heat values. Specific-heat values can be highly inaccurate if the material departs significantly from a low-temperature Debye model.

The large apparent γ arising from the very large changes in elastic constants with temperature that we observe, and the nearidentical fractional temperature dependence of both bulk and shear moduli lead to some new insights into itinerant-electron density. A main theme in explaining actinide-element physical-property oddities is the density of itinerant (free) electrons. Electron density changes systematically as atomic number increases in the actinide series (actinium, thorium, protactinium, uranium, neptunium, plutonium, americium, . . .).



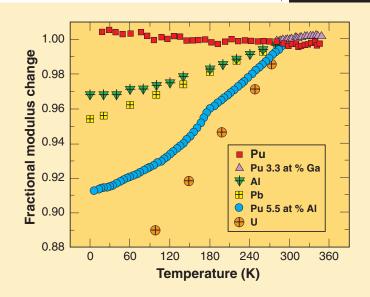
In this photo of a resonant-ultrasoundspectroscopy (RUS) measurement cell, the specimen (a parallelepiped in this case) sits sandwiched between two lead zirconate titunate (PZT) transducers. No bonds or adhesives are used to attach the specimen; it simply rests on the lower transducer while the weight (a few grams) of the upper transducer holds it in place. One transducer sweeps through frequency. The second transducer detects normal-mode (standingwave) resonance frequencies manifested as vibration-amplitude maxima. A common view is that lighter actinides possess more itinerant, bonding electrons that increase elastic stiffness (through the degeneracy pressure), and heavier actinides possess more localized electrons that contribute less to bonding and less to elastic stiffness. Plutonium sits between these limits and possesses mixed itinerant-localized electron character—a balance that depends on temperature, pressure, alloying, and atomic coordination.

In 1936, Fuchs used extended Wigner-Seitz theory to show how itinerant-electron density affects the shear modulus. Gilman (1971) gave a similar relationship for the bulk modulus, using Heisenberg's principle to estimate the electron-gas kinetic energy.

Combining the Fuchs and Gilman equations gives G/B = 5/8 and v = 7/29. Thus, both G and B soften considerably as electrons change from itinerant to localized. The Poisson ratio remains unchanged, independent of ionic charge and ionic radius.

Such similar *B*, *G* behavior (and invariant v behavior) is exceptional. In most inorganic materials, elastic stiffnesses, absent phase transition, change by up to ten percent from 0 to 300 K. Ordinary materials show substantial Poisson-ratio changes. However, if a single physics driver produces larger changes in elastic stiffness with temperature, then these larger changes will overwhelm usual processes, revealing behavior associated primarily with whatever drives the large changes—in this case electron localization during warming.

The figure at right shows our v(T) measurements for α -plutonium (monoclinic) and for δ -plutonium (face-centered cubic) alloyed with aluminum or gallium. To show typical v(T) behavior, we show handbook measurements for aluminum (a typical face-centered cubic metal), lead (a heavy metal often compared with plutonium), and α -uranium (a heavy-metal early actinide). Lead shows an expected relatively large positive slope,



The relative Poisson ratio/temperature for six materials. Aluminum (Al) represents a typical face-centered cubic metal. Lead (Pb), also face-centered cubic, represents a heavier metal, more comparable with plutonium (Pu). Compared with aluminum, lead shows a larger change because of its lower melting point. Uranium (U) (orthorhombic) represents a heavy, actinide metal. It shows a large convex-shape change that reflects its magnetic-state transitions. Alpha (α)-plutonium shows a temperature-invariant Poisson ratio, as predicted for the case where itinerant-to-localized electron transitions dominate the interatomic bonding. The more-dilute delta (δ)-plutoniumgallium (Ga) alloy behaves like α -plutonium, that is, it has a temperature-invariant Poisson ratio, also suggesting itinerantto-localized electron transitions in this material. The moreconcentrated δ-plutonium-aluminum alloy behaves like a typical metal, suggesting that higher alloying completes the itinerant-tolocalized transition. The kink near 180 kelvin (K) shows the onset of the δ - α transformation, estimated to be 5 percent α -phase at zero temperature.

reflecting its low melting temperature. Alphauranium shows an even larger (and convex) positive slope, arising probably from its well-known magnetic interactions, which always alter elastic responses. Alpha-plutonium shows small, nearly temperature-invariant, changes.



These changes agree with the prediction made by the above simple electrostatics model, suggesting that the physics driver is the degeneracy pressure of the itinerant electrons and that the number of these electrons changes with temperature.

Using the Fuchs-Gilman equations, we can estimate the number of itinerant electrons. Substituting the observed bulk modulus B = 71 GPa for α-plutonium at 0 K, and the atomic radius $r_0 = 1.34 \text{ Å}$ (obtained from handbook unit-cell dimensions), we obtain an ionic charge q = 2.0, meaning that six of α-plutonium's eight outer electrons are localized. We assume that these are the six f-electrons, the ground-state electron configuration being [Rn]5f ⁶7s². (A theory by Savrasov and Kotliar for δ-plutonium gave a 5f ⁵ ground state, and one by Wills et al. gave 5f 4.) Recognizing some arbitrariness in these electron-orbital assignments, we note that plutonium (as evidenced by its low bulk modulus) behaves more like an sp-electron metal than like a d-electron metal; more like lead than like iron.

From the Fuchs-Gilman equations, we can derive a relationship connecting ionic charges at different temperatures:

$$q_2 / q_1 = (B_2 / B_1)^{1/2}.$$
 (5)

Considering the α -phase bulk-modulus change from 0 to 300 K (71 to 55 GPa), we can estimate that, for this temperature interval, q_2 / q_1 = 0.88. Thus, warming from 0 to 300 K converts 0.24 electrons from an itinerant state to a localized state. Invoking this itinerant-localized electron-state change explains the unusually large bulk-modulus decrease with increasing temperature and the invariant Poisson ratio. Here, we neglected the smaller bulk-modulus decrease that arises from usual lattice softening. We assume that the itinerant-localized transition continues to occur as plutonium is warmed above 300 K, perhaps accounting for plutonium's low melting

temperature and multiple (six) crystal structures. Sufficient alloying with gallium or aluminum produces a Poisson ratio with near-typical temperature dependence. All this suggests that electron localization stabilizes the face-centered cubic δ -plutonium phase in the maximally localized state and that delocalization is arrested in cooled gallium-stabilized δ -plutonium.

The figure on page 7 shows a second remarkable result: the Poisson ratios for facecentered cubic high-aluminum-content δ-plutonium-aluminum alloys show near-typical temperature behavior. Thus, sufficient alloying completes localization, blocking changes with temperature. This conclusion finds confirmation in the observation that the bulk modulus of the plutonium-aluminum alloy changes only 13 percent between 0 and 300 K, versus a 30-percent change for unalloyed α -plutonium. Support comes also in the conclusion from theory that the degree of f-electron localization depends strongly on chemical environment, on the interaction between bound f-electrons and band electrons of non-f origin.

Our measured elastic constants, especially the Poisson-ratio results, and our electron-gas interpretation yield the following conclusions: in plutonium all six 5f electrons are nominally localized; as plutonium is warmed, localization increases; in face-centered cubic δ-plutonium, localization reaches completion, whether through increased temperature or through alloying; and theories that include a large itinerant-electron fraction lack support in the observed elastic constants. We can also conclude that the α - δ phase transformation does *not* result from abrupt electron localization; that for both phases, the ionic charge $q \approx 2$; that plutonium behaves more like an sp-electron metal than like a d-electron metal; and that the elastic-constants temperature dependences show no phase-transformation evidence (electronic, magnetic, or structural).

Delta-plutonium's elastic anisotropy:

Another enigma providing interatomic-bonding insights?

bout thirty elements occur as face-centered cubic crystal structures, the closest packing of hard spheres in an infinite space. Common examples include aluminum, copper, silver, gold, and lead. Face-centered cubic elements possess a useful feature: high mechanical ductility arising from easy dislocation movement. The material's elastic constants provide interest in this regard because dislocations move in an anisotropic elastic-strain field.

Face-centered cubic crystal structure of delta (a)-plutonium.

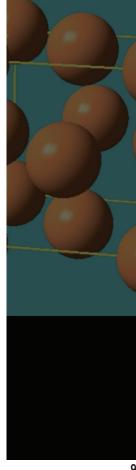
Besides relating to plastic deformation, elastic constants also relate to many other solid-state properties, including atomic-vibration amplitudes, characteristic vibration frequencies (Einstein, Debye), creep, diffusion, fracture strength, hardness, melting, phase transformations, phonon spectra, point-defect energies, sound velocities, stacking-fault energy, theoretical strength, superconducting-transition temperatures, and thermal conductivity. In principle, the elastic constants relate to any lattice-vibrational property.

Among all the face-centered cubic elements, delta (δ)-plutonium shows the most remarkable elastic constants. This extreme behavior appears particularly in the Zener anisotropy ratio $A=2C_{44}$ / $(C_{11}-C_{12})$, the average value being 2.5 ± 0.5 for face-centered cubic elements. For δ -plutonium this ratio is 7.0. This means

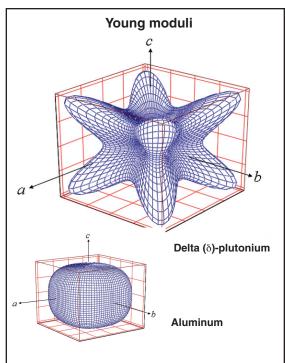
that δ -plutonium's various elastic constants, which are fourth-order tensors, change strongly with crystallographic direction. Delta-plutonium's other elastic oddities include unusually high C_{12}/C_{11} and C_{44}/C_{11} ratios.

A material has many elastic constants; the more common include the Voigt elastic stiffness C_{ij} , Young modulus E_{hkl} , shear modulus $G_{hkl,lmn}$, bulk modulus B, and Poisson ratio $v_{hkl,lmn}$. The bulk modulus lacks indices because, being the negative ratio of pressure change to fractional volume change, it is a rotation-invariant scalar. The Poisson ratio is not a tensor, but instead the dimensionless ratio of elements of two fourth-order tensors.

At Los Alamos, we focus on plutonium's elastic constants because they provide accurate core physical properties that relate directly to theory and to other core physical properties (enumerated above). We measure them by various methods, especially resonance-ultrasound spectroscopy (RUS), which is described in the preceeding article. This article was contributed by Hassel Ledbetter of the **Nuclear Materials** Technology Division; Albert Migliori of the Materials Science and Technology **Division-National High Magnetic** Field Laboratory and the Seaborg Institute; and Robert G. Leisure, Physics Department, Colorado State University, and visiting scientist at the NHMFL.

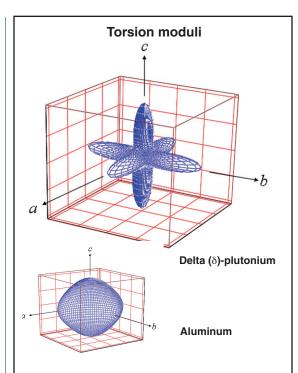


DELTA-PLUTONIUM'S ELASTIC ANISOTROPY



Young moduli of delta (a)-plutonium and aluminum. Aluminum shows near isotropy. Plutonium represents the most anisotropic cubic element, a property not yet well understood. Maxima occur along <111>, minima along <100>.

The diagrams above compare δ -plutonium, the most anisotropic known element, with aluminum, a more-typical material. Plutonium's Young modulus shows eight lobes along the four <111> directions with minima along the six <100> directions, the maximum/minimum ratio being 5.4. The contrast with aluminum is enormous where this ratio is 1.3. Plutonium's torsion modulus shows opposite behavior to the Young modulus with maxima along the <100> directions and minima along the <111> directions. The maximum/minimum ratio is 5.0; for aluminum this ratio is 1.2.



Torsion moduli of delta (a)-plutonium and aluminum. Plutonium's maxima occur along <100>, the minima directions for the Young modulus. The plutonium figure, reflecting elastic compliance more than elastic stiffness, presents a surprise.

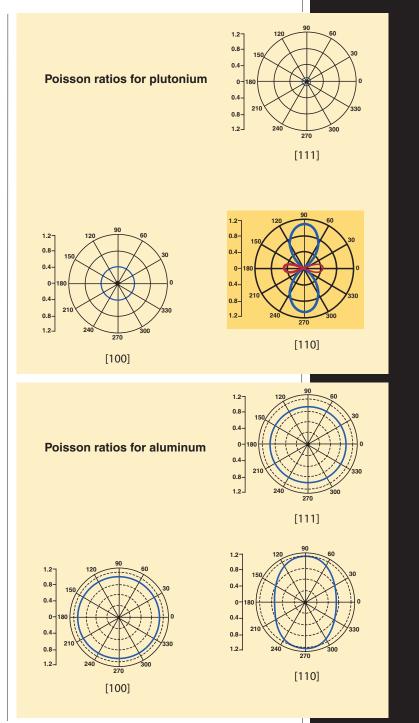
Plutonium's Poisson ratio provides particular interest because it shows different profiles in different principal crystallographic directions. And, in some directions, it shows large negative values. A negative Poisson ratio means that pulling in one direction causes expansion in the transverse plane, a phenomenon associated traditionally with cork and network structures.

Results for plutonium are shown with those for aluminum in the illustrations at right. The illustrations are for extensional strains in the three crystalline directions <100>, <110>, and <111>. For plutonium, an extensional strain e in the <100> direction results in an isotropic contraction of 0.42e in the perpendicular direction. For an extension e in the <111> direction, the perpendicular isotropic contraction is only 0.09e. For an extension e in the <110> direction, the lateral strain is highly anisotropic. In fact, the extension e in the <110> direction results in an extension 0.48e in the <-110> and <1-10> directions, but a contraction of 1.09e in the <001> direction.

Much current research proceeds on negative-Poisson-ratio materials, a material class called auxetic. The diagrams for plutonium dispel any notions of elastic similarity, isotropic behavior, always-positive Poisson ratios, and simple crystallographic-direction/stiffness relationships.

Plutonium's extreme high elastic anisotropy reveals interesting features of its interatomic bonding. Max Born's lattice-dynamics model (1943) for face-centered cubic crystals predicts A=2. Invoking farther-neighbor interactions or volume forces cannot increase this by much. Thus, we need to invoke angular (three-body) forces, which impose difficult computational problems. Fuchs' (1936) extended Wigner-Seitz calculations showed that for face-centered cubic lattices, considering only the electron-electron-ion electrostatic terms, one obtains A=9.0, thus providing a promising possible explanation for δ -plutonium's high elastic anisotropy.

Poisson ratios for delta (a)-plutonium and aluminum along three principal crystallographic directions. Along [100], plutonium shows negative (red) values. This means unusual (auxetic) behavior: opposite sign for the transverse strain.



DELTA-PLUTONIUM'S ELASTIC ANISOTROPY

> The high anisotropy also means that δ -plutonium lies near a Born mechanical instability boundary. Well known are the Bain lattice correspondence δ – δ' – ϵ phase interrelationships among face-centered cubic, face-centered tetragonal (body-centered tetragonal), and body-centered cubic phases. The high elastic anisotropy and the low C_{II} – C_{I2} value mean that these three crystalline phases interconvert easily, a fact first appreciated by Zener for the alkali metals. (Body-centered cubic at ambient temperatures, only the alkali-metal elements rival plutonium's high elastic anisotropy.) Zener also realized that high elastic anisotropy means high vibrational entropy, lower free energy, and a tendency toward phase transformation.

Zener's elastic-anisotropy values for several metals follow:

Aluminum	1.22
Gold	2.85
Silver	2.88
Copper	3.16
Lead	4.07
δ-plutonium	7.03

Clearly, for most purposes, aluminum can be treated as a nearly isotropic material. Plutonium's enormous elastic anisotropy implies strong anisotropy in related mechanical-physical properties, and it provides a keen

quantitative check (constraint) on the many emerging ab initio theories of plutonium.

Our current research focuses not only on plutonium's peculiar anisotropic elastic properties, but also on how these properties depend on composition, temperature, and pressure, where another set of peculiarities appear. Recently, we succeeded in relating the elastic constants to 5f-electron itineracy localization, a problem affecting the physical properties of all the actinides.

Further reading:

"Elastic properties of face-centered cubic plutonium," Hassel Ledbetter and Roger Moment, Acta Metallurgica 24 (1976), pages 891-899.

Elasticity and Anelasticity of Metals, Clarence Zener (University of Chicago Press, Chicago, 1948), especially Chapters II-IV.

"Lattice theory of mechanical and thermal properties of crystals," Günther Leibfried, in *Handbuch der Physik* (Springer, Berlin, 1955), pages 104-324.

Pioneer actinide chemist **Larned Asprey dies**



Larned "Larry" B. Asprey, one of the giants of actinide and fluorine chemistry, died March 6, 2005, in Mesilla Park, N.M. He would have been 86 on March 19. He retired from Los Alamos National Laboratory in 1986 after conducting more than thirty-five years of fundamental and applied research, which was critical to the present-day understanding of the chemistry of actinides and lanthanides, as well as aspects of main-group elements.

Asprey published approximately 150 key peer-reviewed papers and held eight patents, in addition to issuing countless technical reports. His formal recognitions include the Glenn T. Seaborg Actinide Separations Award, New Mexico Chemist of the Year, Los Alamos Distinguished Service Award, and an invited one-year appointment at the Karlsruhe Nuclear Research Center in Germany. He was active in the Inorganic Division of the American Chemical Society, serving as secretary-treasurer, councilor, member of the Executive Committee, and organizer of technical sessions.

A paper co-authored by Bob Penneman on transuranic chemistry was selected for the groundbreaking first Atoms for Peace conference in Geneva in 1954, where actinide chemistry

Larry Asprey was both a co-worker and friend. He had a clear and focused view of his path and pursued it relentlessly, hurdling obstacles that would deter many. Early on, he stated that he wanted to spend his time in the lab and to be shielded from administrative work. As his section and later group leader, my role was his "supervisor/helper" for thirty-five years. To get the best from Larry I had only to make a suggestion, which he would immediately attack and substitute with a better one.

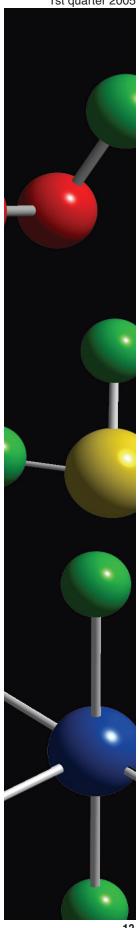
Larry flourished in the laboratory, using a variety of exotic and hazardous materials. They spanned the radioactive actinide elements from protactinium through californium and the potent oxidizers, chlorine trifluoride (CIF₃), dioxygen difluoride (O₂F₂), and fluorine.

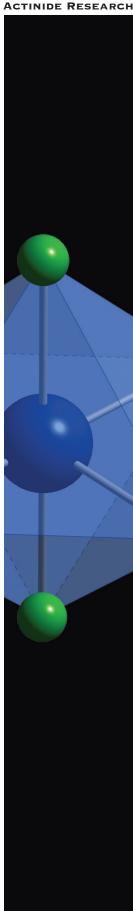
His work with such materials often led to adventures like the time a group of researchers wanted to measure the velocity of sound in chlorine trifluoride. Larry cautioned them that the cell they needed to provide had to be scrupulously clean. The cell was filled at DP Site and placed in a temperature-controlled silicone oil bath. We were in an adjacent room when we heard a low rumbling that became increasingly louder. It turns out that a tapped screw hole in the cell had not been entirely cleaned of machine oil. The chlorine trifluoride reacted with the oil, ate through the cell, and began reacting violently with the silicone oil bath. When the fume hood quit dancing, the reaction stopped, and our wide-eyed visitors rethought their participation.

-Bob Penneman, retired

was discussed for the first time at an open international setting. That meeting marked the first interactions with Soviet scientists working in the actinide area.

In addition to his prolific and influential publication record, Asprey had a profoundly positive influence on his peers as well as younger practitioners of actinide and fluorine chemistry. He was always there to serve as professional





mentor, personal friend, confidant, and fellow adventurer in the outdoors he loved. His legacy of impeccable scientific integrity, zest for hard work and play, humanity, and sense of fair play has been continued by numerous former students and collaborators now working at Los Alamos, in academia, and elsewhere. Many young staff and students were made to feel part of the wonderful Asprey family, which included his wife of more than 60 years, Margaret "Marge" Asprey, and seven children.

Asprey's legendary skills as a preparative chemist made him highly sought after as a co-investigator. His unique ability to carry out difficult syntheses was often used by other scientists to make critical contributions to broad areas of inorganic chemistry. This body of work ranged from fundamental studies of interest to the academic community to applied programs involving international security issues of the highest magnitude.

Asprey was a brilliant, inspiring, and daring experimental chemist who obtained his B.S. at Iowa State University in 1940. His work in actinide chemistry began in early 1944 during his Army enlistment. He was assigned to the Manhattan Project's effort to separate and purify plutonium under Glenn T. Seaborg at the University of Chicago's Metallurgical Laboratory and

As a new summer student in June 1950, I was assigned to Bob Penneman's CMR-4 Group. Larry Asprey and Bob were the principal researchers into the then barely known intricacies of actinide chemistry. It was a wonderful experience for a young graduate student. While I'd had a course in radiochemistry, I had only dealt with small tracer amounts of activity, and that summer we were dealing with multi-milligram quantities of pure americium-241.

Back then, the word "mentor" wasn't in such common use as it is today, but Larry was a wonderful mentor. He was always willing to take the time to describe details of experiment planning and obvious precautions to take. Larry's experience with americium while working at Berkeley was a great asset at Los Alamos. He seemed to have a clairvoyant insight into basic plutonium, americium, and curium chemistry so that his shrewd guesses usually turned into scientific fact. I'm so very proud to have known him all the years since my first months at Los Alamos, and I'm even more proud that several of my publications list Asprey and Keenan as co-authors. His effervescent enthusiasm for actinide research was matched only by his love of the outdoors. Weekend fishing, hunting, or backpacking trips were exhilarating breaks from the laboratory.

—Tom Keenan, retired

I worked with Larry for many years, and he was a great collaborator. We used infrared spectroscopy (IR) to study molecules isolated in low-temperature matrices of solid argon. Together we studied the spectra and determined force constants for many interesting molecules, including nitrosyl fluoride and chloride (ONF and ONCI) with isotopic mixtures of oxygen(16+18) and nitrogen(14+15) and chlorine(35+37). Later we made detailed studies of uranium hexafluoride (UF_e) and uranium pentafluoride (UF₅) isolated in solid argon. We also studied IR spectra of pure uranium hexafluoride in the gas and solid phase at very low temperatures. References to our collaborative efforts can be found in the book Inorganic Vibrational Spectroscopy by L.H. Jones, 1971, Marcel Dekker Inc.. New York.

> —Lew Jones, Laboratory Fellow, retired

quickly became an important member of the Plutonium Recovery Section. From this work, a patent application involving tributylphosphate was filed on May 8, 1947, with H.H. Anderson as lead author and Asprey as second author. This work formed the basis of the PUREX (plutonium uranium extraction) process, still used worldwide to recover plutonium. Asprey's Manhattan Project work signaled a highly auspicious beginning to a career-long involvement with the lanthanide and actinide elements.

After World War II, Asprey obtained his Ph.D. in 1949 from the University of California–Berkeley under the tutelage of the noted experimentalist B.B. Cunningham, who had isolated the first americium compounds. Asprey's thesis, "Equilibria in the Oxide Systems of Praseodymium and Americium," explored the extremely complex nature of actinide and lanthanide oxides, an area of actinide research still of interest as highlighted in recent issues of *Actinide Research Quarterly*.

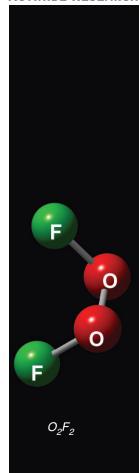
Asprey joined Los Alamos Scientific Laboratory in 1949 and continued to develop the fundamental and applied chemistry of the actinides. He participated in the first sub-gram and gram-scale isolation of americium and the elucidation of its chemistry, then largely unexplored. Small amounts of plutonium-241, which decays to americium-241, are formed together with plutonium-239 in neutronirradiated uranium. Plutonium metal scrap from machining slugs for Los Alamos' Clementine reactor contained about 50 parts per million americium-241 available for americium recovery. Concentrated salt wastes with a 300:1 ratio of lanthanide to americium, but very dilute in americium, also were available from purification of

Preparative chemistry is the foundation of most chemical research and in this arena Larry was universally recognized as a master. His ability to carry out difficult reduction syntheses using esoteric techniques designed for handling dangerously unstable, toxic, or highly radioactive materials was legendary. It was these skills that made him the "go-to guy" for many talented co-investigators who were interested in doing physical studies including spectroscopic or structural investigations. In a real sense he enabled a large body of work outside his core research efforts.

In the late 1960s when I joined the group that eventually became INC-4, Larry was the kernel of a small but highly productive collection of synthetic chemists. Over the span of the next nearly three decades the synthesis effort in that group became increasingly important and, as the group expanded, formed the basis for a dazzling proliferation of programs, a list of which would span most of the interests of modern chemistry. These programs were a strong drawing card that attracted some of the country's very best young chemists to Los Alamos and, indeed, many of them now lead key efforts or hold key management positions in chemical programs throughout the Laboratory.

Over the years many of the younger staff members had direct interactions with Larry and found his dedication and enthusiasm for chemistry to be contagious and inspirational. He was extremely generous with his time and talents inside and outside the Laboratory and had the habit of inviting us all to join him in many outdoor adventures in Northern New Mexico. Indeed, he made us feel that we were honorary members of his wonderful family. Those of us who spent all or part of our careers serving a Laboratory whose success we felt was critical to the national welfare will remember Larry with a deep sense of gratitude.

-Bob Ryan, retired



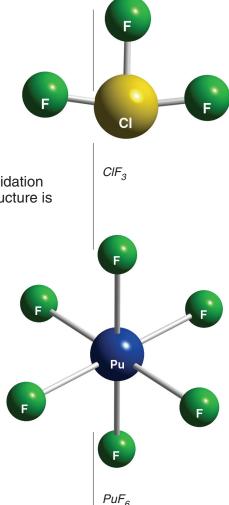
Hanford plutonium nitrate. Separations processes were successfully developed and americium was isolated on the gram scale for the first time from these very lean sources.

Asprey's important early Los Alamos contributions to elucidating the basic chemistry of americium are documented in benchmark publications with his co-workers. This body of work includes discussions of the disproportionation of americium(V), the direct oxidation of americium(III) to (VI), the measurement of the americium(V)/(VI) oxidation potential, the demonstration that the linear uranyl structure is maintained in americium(VI), the demonstration of the elusive divalent state in the di-iodide, the discovery of the tetravalent state in fluoride salts, and the first absorption spectrum of a soluble and relatively stable americium(IV) species in

concentrated "aqueous" fluoride solution. In subsequent work, Asprey and co-workers demonstrated that curium, at the center of the 5f series, could be oxidized to curium(IV). His prolific investigations of unusual oxidation states of americium and curium were extended to berkelium. californium, and lanthanide elements. Observation of the fluoride solubility of americium(IV) allowed berkelium fluoride to be readily separated from its americium bombardment target, and was critical to the first observation of this new element by Ghiorso and co-workers. This portion of Asprey's research also includes the first demonstration of the tetravalency of berkelium and californium, the preparation of the first divalent thulium compounds and two new tetravalent lanthanides: neodymium and dysprosium.

Asprey's work on protactinium, americium, berkelium, and californium metal preparation was also highly influential. Actinide metals prepared by him were indispensable for structural, magnetic, and superconductivity studies of these metals. This work also included the first distillation of americium—a matter of significant practical and fundamental importance.

Collaboration between Asprey and the great crystallographer William H. Zachariasen began in 1954 and continued for some twenty years. (Zachariasen's work is highlighted in ARQ 2nd quarter, 2004.) As an interesting example of this collaboration, Zachariasen recognized the x-ray pattern of the pentavalent



americium compound, KAmO₂CO₃, prepared by Asprey and co-workers, as being essentially identical to that of a putative hexavalent plutonium compound of the 1940s, that "the chemists" claimed was KPuO₂(OH)₃. Based on the correct formula, Zachariasen deduced immediately that the original plutonium compound really was KPuO₂CO₃ with pentavalent plutonium, both formulas having the required five oxygens, but with carbonate instead of the proposed three hydroxyls.

Asprey became a noted fluorine chemistry authority through his work on high-oxidation-state chemistry of the actinides and lanthanides. He discovered and patented the process still used to prepare extremely pure elemental fluorine. His fluorine expertise was used to prepare many volatile transition metal, main group, and actinide fluorides for important collaborations in vibrational spectroscopic studies in gas and low-temperature matrix isolation phases. This influential body of work is among the most heavily cited of Asprey's publications and contributed significantly to the major national effort on laser-based isotope separation methods. Related frequently cited work includes publications defining fundamental structure and bonding properties of simple molecular fluorides in solution, gas, and solid phases.

In the last years of his career, Asprey became heavily involved with Los Alamos research on the superoxidizers dioxygen difluoride (${\rm O_2F_2}$) and krypton difluoride (${\rm KrF_2}$), which can convert plutonium substrates to volatile plutonium hexafluoride (${\rm PuF_6}$) at room temperature. Because of the practical implications of this observation, preparatory methods were needed for unprecedented quantities of these difficult reagents. Asprey was key to success in this superoxidizer work, as illustrated by his co-authorship of a burst of publications on the preparation and application of these remarkable compounds.

Ever the scientist, Asprey bequeathed his body to the University of New Mexico Medical School. A scholarship fund has been set up in his name at New Mexico State University. Donations made out to "NMSU Foundation" may be sent to: The NMSU Foundation, in memory of Larry Asprey, P.O. Box 3590, Las Cruces, NM 88003.

Larry Asprey strongly touched all those who were fortunate enough to work and play with him. Those who knew this remarkable man will remember his remarkable life fondly. A quote by Emile Zola—"I am here to live out loud!"—may be an appropriate description of the life and contributions of Larry Asprey, both as a scientist and as an individual.

-P. Gary Eller, Bob Penneman, and Bob Ryan

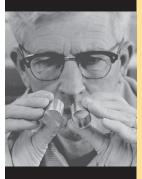
In the previous tributes, it is abundantly clear that Larry Asprey was an eminent actinide chemist. However, it would be incomplete to stop there. In a broader picture, it can be more completely stated that Larry Asprey was an outstanding inorganic synthetic chemist who made creative advances not only in the chemistry of the actinide elements, but also in the synthesis of new main-group element species as well as transition metal compounds. Most notably, he was an internationally recognized fluorine chemist. The fields of actinide and fluorine chemistry regularly intermingled in Larry's hands, but he also made numerous contributions in the larger context of the fundamental reactivity of fluorine and fluorine compounds important to Los Alamos technical objectives and to fundamental inorganic chemistry.

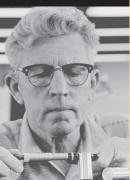
As a postdoctoral student who studied with Larry in the early 1970s, I would also like to say that it would be even more incomplete to just pay tribute to Larry Asprey's technical accomplishments. Larry was much more than a fine chemist. He was an outstanding teacher, mentor, friend, and confidant. Although it is a cliché, it is accurate to say that Larry gave the shirt off his back to those who studied with him. We spent many hours together in the laboratory and we shared a good deal of time on activities outside the formal laboratory setting.

My wife and I truly were made to feel part of the Asprey family and heaven knows Marge and Larry did not need more kids to look after! We did a lot of nice chemistry together but my mosttreasured memories of Larry Asprey will be of the person. The impacts that he had in shaping my own subsequent professional and personal life are large and are highly valued by me. I am sure that all of the young chemists who worked with Larry would share this same sentiment. He will be missed but certainly remembered by all of us as we do our own mentoring of the next generation.

> —Bob Paine, University of New Mexico

Art Beaumont compares a mockup welded capsule and the gauge tube into which a heat source capsule had to fit to meet size specifications.





Beaumont measures the outside diameter of a gauge tube with a micrometer. The gauge tube was designed to ensure that encapsulated heat sources would fit inside the available space in the artificial heart unit.

From heat sources to heart sources:

Los Alamos made material for plutonium-powered pumper

Be still my beating heart. In a novel program that started in the 1960s, the Laboratory began a project to help those faint of heart. In 1967 Los Alamos Scientific Laboratory explored a new mission: developing a self-contained energy source that would last for decades to power a conceptual artificial heart. The energy source would be powered by the same material developed in 1963 for the space program—plutonium-238. The heat from the radioactive decay of the plutonium-238 can readily be used directly or used to produce electric power for space probes, etc.

In a joint effort between the National Heart and Lung Institute (NHLI) and the Atomic Energy Commission (AEC), the Department of Energy's predecessor agency, Los Alamos researchers began the endeavor to balance the hazards of plutonium-238 with the benefits. Researchers believed that they could minimize the element's neutron radiation effects while supplying future artificial heart recipients with a long-life power source.

ing future artificial heart recipients with a long-life power source.

Members of Chemistry-Metallurgy "Baker" Division's CMB-11 fabricated the source with 50 watts of energy—enough to drive the artificial heart—and focused on reducing the radiation while George Matlack and Joe

Bubernak of CMB-1 analyzed the radiation properties.

Researchers considered two or three different isotopes but finally chose plutonium-238 because of its half-life of eighty-seven and a half years, which was long enough to provide power with no significant loss of energy during the lifetime of the mechanical heart. The idea came from the Lab's active space program. More than thirty years ago, NHLI concluded that an external power source needed to run the implanted device would be as large as a telephone booth, somewhat impractical for artificial heart recipients.

Scientists at Los Alamos believed that they could make a plutonium-238 heat source small enough to implant into the human body. The heat source would power a stirling-cycle engine that would pump the blood. But the conundrum was if the plutonium-238 was powerful and long-lasting enough to save the patient, would the radiation effects end up killing the heart recipient?

Another drawback to using plutonium-238 metal was that it has a relatively low melting point, so if a deceased patient was cremated, the crematorium might become a radiological cleanup site. The research team considered using an alternative form with 3 percent gallium, which raised the melting point, but it still didn't prevent the melt that could occur in a crematorium.

Researchers experimented with plutonium-238 oxide from Savannah River, but the radiation levels were hard to measure at the time, and several light-element impurities produced an alpha-neutron reaction.

Plutonium-238 is relatively easy to shield because its gamma rays are mostly of low energy and it produces a low amount of spontaneous neutrons. However, in the presence of other light-element isotopes such as nitrogen-14, oxygen-17, oxygen-18, carbon-12, or fluorine-19, the neutron emission is much higher because of the interaction of alpha particles (produced by plutonium-238 radioactive decay) with the other elements. A heavy but energetic alpha particle (helium ion), which can be shielded with a piece of paper, would hit an atom like fluorine and cause a neutron to escape. Neutrons are very difficult to attenuate and cause significant potential radiological doses to employees and candidate artificial heart recipients.

This article was contributed by Kathy DeLucas of the Public Affairs Office; Jim Foxx of the Nuclear Materials Technology Division; and Robert Nance, formerly with the Chemistry-Metallurgy "Baker" Division and now retired.



Larry Mullins records data during a directoxide reduction (DOR) experiment.

Scientists reduced this threat of the induced neutrons by two steps. In the original process, the first step was to convert the plutonium oxide to metal, which was historically done by means of a plutonium-fluoride intermediate with excessive alpha-induced neutrons. From the health-physics standpoint, this was quite undesirable for workers in the immediate area.

Researchers therefore came up with the direct-oxide reduction process. In this process the plutonium oxide is mixed with calcium chloride-calcium fluoride or only calcium chloride, along with calcium metal, and heated to produce plutonium metal. A variation of the direct-oxide reduction (DOR) process today is the mainstay of producing plutonium-239 for the weapons program.

Researchers prepared the metal and removed nonradioactive light elements from it through electrorefining. The pure metal ingot was then converted to finely divided metal particles by reacting it with hydrogen and subsequently removing the hydrogen three times—a hydride-dehydride cycle. The purified finely divided metal was then ready for reaction with oxygen-16 water vapor, in the second step.

Normal water contains three oxygen isotopes: -16, -17, and -18. The oxygen-17 and -18 isotopes are susceptible to alpha-neutron reactions, but the oxygen-16 isotope is not. Highpurity water vapor enriched at Los Alamos in the oxygen-16 isotope was reacted with the finely divided plutonium-238 metal to prepare plutonium oxide that had minimal alphainduced neutron radiation. The techniques for the hydride-dehydride cycle and preparation of the plutonium-238 oxygen-16 oxide fuel were developed by Robert Nance.

The process of reacting oxygen-16 water with plutonium oxide to produce isotopic exchange is still used today to reduce the alphaneutron reactions in heat sources for the space program. The recently highly successful Cassini space probe to Saturn and its moon, Titan, is a good example. A number of earlier, spectacular, landmark deep-space explorations such as Voyagers I and II were powered by specially processed plutonium-238.

Because Congress was funding dual-track research programs, it decided to proceed with NHLI research, so the AEC's program was dropped and the project ended in 1977.



Carl Peterson transfers plutonium-238 oxygen-16 oxide inside an inert-atmosphere glove box from a storage container in preparation for pressing a fuel pellet. The furnace in front of Peterson was used for sintering pressed pellets at high temperature.

Even if the project had continued, there were some real challenges to overcome. Insurance companies surely would have balked at the \$200,000 price tag. The plutonium was encapsulated with three layers of metals: first tantalum, then tantalum-10 tungsten, and finally an outermost capsule of platinum-20 rhodium. These metals provided shielding and protected the heat source from oxygen. Three of the "D" cell sized 50-watt plutonium heat sources were made for testing.

Another drawback was discovered when rigorous tests were conducted. Researchers found that the encapsulation would not survive a 30.06 gunshot, which could cause a radioactive contamination threat if a recipient was shot through the heart. Finally, there was a problem at that time identifying pump materials that would not cause coagulation during long-term contact with blood.

While the program demonstrated many firsts at the Lab, some of which are still used in actinide processing today, there was also a successful spinoff. The Lab produced 63 grams of high-purity plutonium-238 metal for the pacemaker program. Medtronics made about 250 of the plutonium-powered pacemakers, and about twnty-two were still stimulating human heart more than twenty-five years after they were manufactured, a feat that no battery-powered pacemaker could match. The Nuclear Regulatory Commission maintains strict guidance for hospitals conducting patient monitoring and how to dispose of the pacemaker when the patient no longer needs it. The pacemaker is clearly stamped with the radioactive symbol and is labeled as containing plutonium-238.

Other successful technologies that are still used today as a result of the program include the minimization of neutron radiation and the methods of accurately measuring the radiation from plutonium-238 materials.

Jim Foxx (below left) and Larry Mullins discuss a heliumrelease experiment with a vented plutonium-238 oxide heat source. The encapsulated heat source was welded into a container like the one shown and connected to a helium leak detector by the tube joined to the top of the capsule to measure helium released by the source. The helium was generated by the alpha decay of the plutonium. The experimental apparatus can be seen in the hood behind the experimenters.





The heat source capsule had to be handled inside a glove box with forceps because of the high temperature of the unit. The heat was generated by the alpha radioactive decay of the plutonium-238.

This article was contributed by Randy Erickson and David Alberstein of the Nuclear Materials Technology

Division.



An FS-47 container.

In support of NNSA surplus plutonium disposition program

Los Alamos meets deadline for plutonium oxide packaging and shipping to France

Trom October 2001 to June 2004, Los Alamos researchers purified plutonium oxide for use in fabrication of mixed oxide (MOX) lead assemblies. This work was conducted in support of the National Nuclear Security Administration (NNSA) Office of Fissile Materials Disposition (NA-26) surplus plutonium disposition program. This program is part of a major U.S./ Russian bilateral effort to dispose of surplus weapons materials. The lead assembly effort required negotiations and close coordination among the governments of the United States, France, the United Kingdom, and Japan, as well as the involvement of the European Commission. Many agencies within these governments and numerous U.S. and international contractor organizations participated in the lead assembly effort.

At the time operations were suspended at Los Alamos on July 16, 2004, purification of the material had been completed, and the material had been placed into DOE Standard 3013 containers for shipment to France, where the lead assemblies were to be fabricated. Work remaining to be done consisted primarily of packaging the 3013 containers into Nuclear Regulatory Commission- and Department of Transportation-approved Type B shipping packages designed and fabricated in France, and loading the packages onto DOE Office of Science and Technology (OST) trucks for shipment off site. Arrangements for other steps in the shipping process (OST transport, ocean transport, and land transport in France) were dependent on Los Alamos completing the first part of the shipping process as scheduled.

Shipment of the material from Los Alamos was time critical because the facility in France at which the lead assemblies were fabricated will be completely shut down soon. If the material was not received in France on the committed schedule, there would not be

sufficient time to fabricate the assemblies before shut down of the facility begins. In this event, lead assemblies would not be available until the MOX Fuel Fabrication Facility to be constructed at Savannah River is operational. This would delay implementation of the U.S. plutonium disposition effort by three to five years, increase the cost of the U.S. program by \$1 billion, place the viability of the bilateral program in jeopardy, and compromise U.S. credibility in this and other nonproliferation programs. There were no other alternatives available to NNSA. If the material did not leave Los Alamos on schedule, severe programmatic impacts would have resulted.

Accordingly, the plutonium oxide packaging and shipping activities were separated from other Laboratory activities and were made the focus of an intense readiness verification process before resumption of the work. Working closely with the staff of the NNSA Los Alamos Site Office, Los Alamos staff from six divisions prepared and reviewed procedures, authorization-basis documents, training documentation, and other documents to support this effort.

Because of this detailed preparation effort, as well as the dedication of many Los Alamos staff members, the plutonium oxide packaging and shipping effort was completed on schedule and was conducted safely, securely, and in compliance with all relevant requirements. Fabrication of the lead assemblies was completed in France in early March of this year, and the assemblies will be returned to the U.S. on time for insertion and testing in a commercial nuclear reactor. This effort demonstrates Los Alamos' ability to combine excellence in conduct of operations with excellence in science and engineering.

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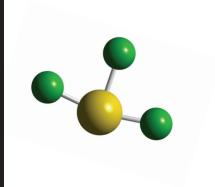
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